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THE UNIVERSITY OF MICHIGAN
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Final Report

COLLECTION AND ANALYSIS OF UPPER-AIR SAMPLES

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Project 2232

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ABSTRACT

A new selective adsorption micro gas analyzer capable of measuring the helium and neon in 0.01 cc NTP of air was completed and successfully operated. Improvements in the construction and preparation of upper-air sampling bottles were made. The instrumentation for sampling at the peak of an Aerobee rocket trajectory was redesigned. An analysis of the possibility of "separation" results being caused by a flow phenomenon was continued.

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1. INTRODUCTION

This is the fifth and final report in a series on Contract No. DA-36-039-SC-56737 describing an experimental program of collecting and analyzing upper-air samples. The work is a continuation of one phase of a program of upper-air research which has been carried out since 1946 by the University for the Meteorological Branch of the Signal Corps. The other phase of the work, that of measuring pressure, density, temperature and winds, is being continued on Contract No. DA-36-039-SC-64659. The sampling work will now be continued as a task under the latter contract. For background material, the reader is referred to the Final Reports of Contracts W-36-039-SC-32307, DA-36-039-SC-125, and DA-36-039-SC-15443.

2. PURPOSE

The purpose of the research is given in Signal Corps Technical Requirements SCL-2370 of 19 January 1954 as follows:

"This specification covers the research into the necessary techniques for the collection and analysis of air samples in the region of 30 to 100 km altitude and a continuing review of the field of upper-air research for the purpose of keeping in contact with work of interest to the Signal Corps.

"The techniques shall be confirmed by field experiments using Aerobee or other rockets as vehicles. Emphasis shall be placed on the following experiments:

"(a) The analysis of upper air and control samples using gas adsorption and/or other techniques.

"(b) The collection of samples in the region 30 to 100 kilometers.

"(c) The performance of subsidiary experiments, reduction of data, calculation of results and preparation of reports."

3. NEW ANALYZER

The measurement of diffusive separation in the upper atmosphere by rocket sampling and the interpretation of the results is summarized in the Final Report of Contract No. DA-36-039-SC-15443. The essence of the situation is that separation has been measured in samples taken between 60 and 100 kilometers but that uncertainties in the experimental method exist. It was decided that these uncertainties could be removed by further sampling and analysis. It was felt also, in the light of the shutdown of the analysis facility at the University of Durham, that a new analyzer should be built at Michigan to insure analysis of upper-air and control samples. Under the previous contract, the analyzer design was determined and construction of some components started, so the major effort on the current contract has been assembly and test. Figures 1 and 2, from a previous report, show the completed analyzer.

The construction and operation of the analyzer worked out about as planned. The use of a miniaturized soft-glass system has eliminated the necessity for a large sample and speedy analysis which were drawbacks of the first Michigan analyzer. The small overall size, which permitted the surrounding of the analyzer with an oven for rapid bake-out, also proved advantageous. The experience gained with the first Michigan equipment also led to an efficient arrangement of controls and pumps so that the new analyzer is relatively easy to operate. The major difficulties centered around three of the most important elements of the analyzer: the transfer pump, the fractionating column, and the output gage.

3.1 THE TRANSFER PUMP

One of the time-consuming operations of an analysis is the transfer of the sample from the sampling bottle to the analyzer. In the Durham and the first Michigan analyzers, this was accomplished with large (800 cc) Toepler pumps arranged for automatic operation. In the case of a bottle which sampled at 70 km, enough air for analysis can be transferred in an hour. In the case of 100-km air, all of the sample is required for one or two analyses and this transfer may require as much as 8 hours of pumping. It was thought desirable, therefore, to use a more efficient pump. Various pumps were considered: mechanical displacement, Archimedes, carrier-gas Toepler, jet, diffusion, and charcoal trap. The last three were tried in various forms and rejected as liable to break or as no more efficient than a Toepler. After some effort was expended on these developments, it was concluded that, although a faster pump probably could be adapted to the analyzer, the advantage to be gained was not worth the effort. Therefore, a

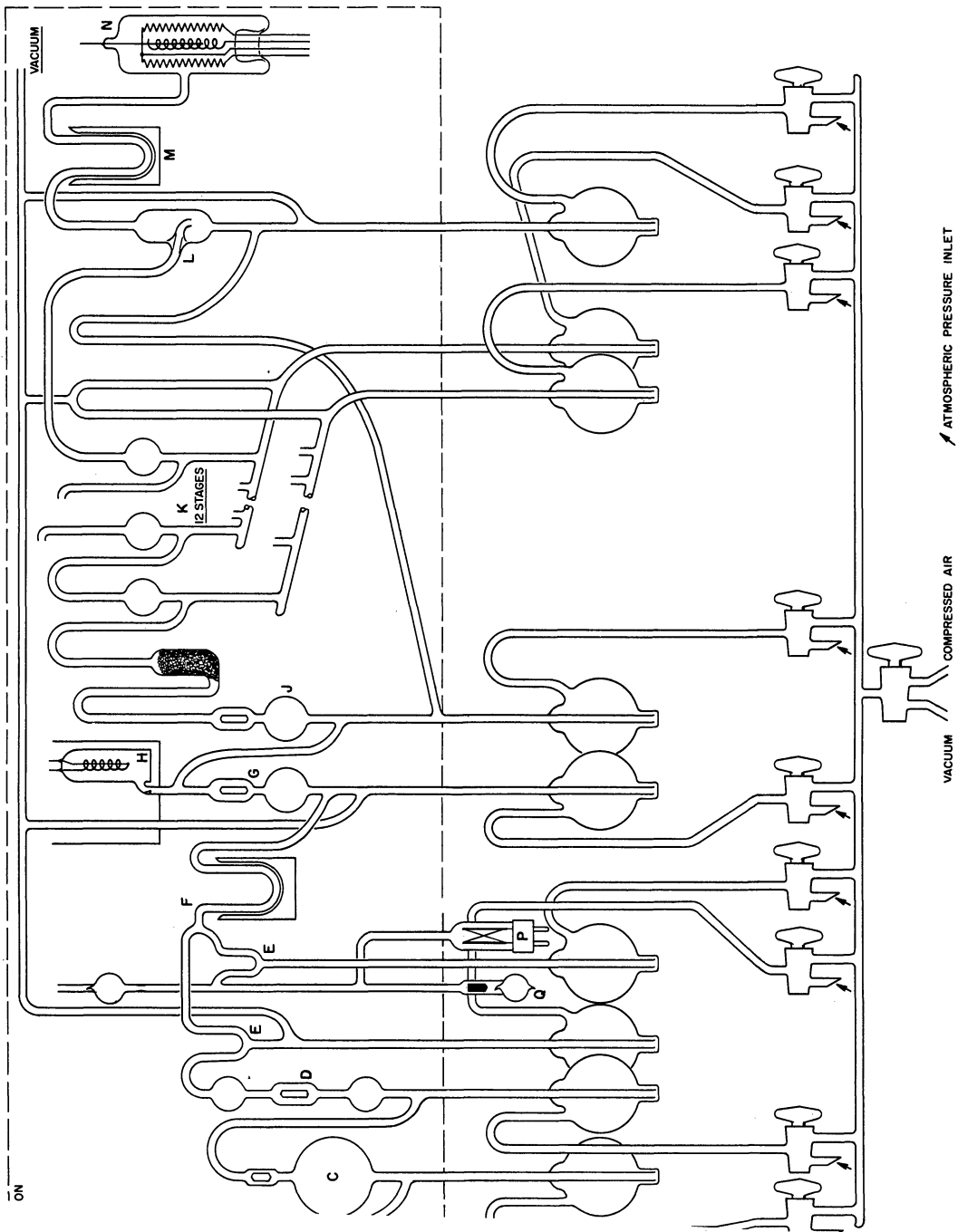


Fig. 1. Schematic of New Analyzer. A. Sample, B. Pirani Gage for Bottle Pressure, C. Large Toepler Pump, D. Small Toepler and Storage Vessel, E. Mercury Cut-Offs, F. CO₂ Cold Trap, G. Oxygen Cell Toepler, H. Oxygen Cell, J. Column Toepler, K. Fractionating Column, L. Pipette, M. Ion Gage Cold Trap, N. Ion Gage, P. System Pirani Gage, Q. Control Sample Vial, R. Breaker. Mercury containers and stopcocks in the lower part of the diagram are for operating the pumps, valves, or column to which they are connected.

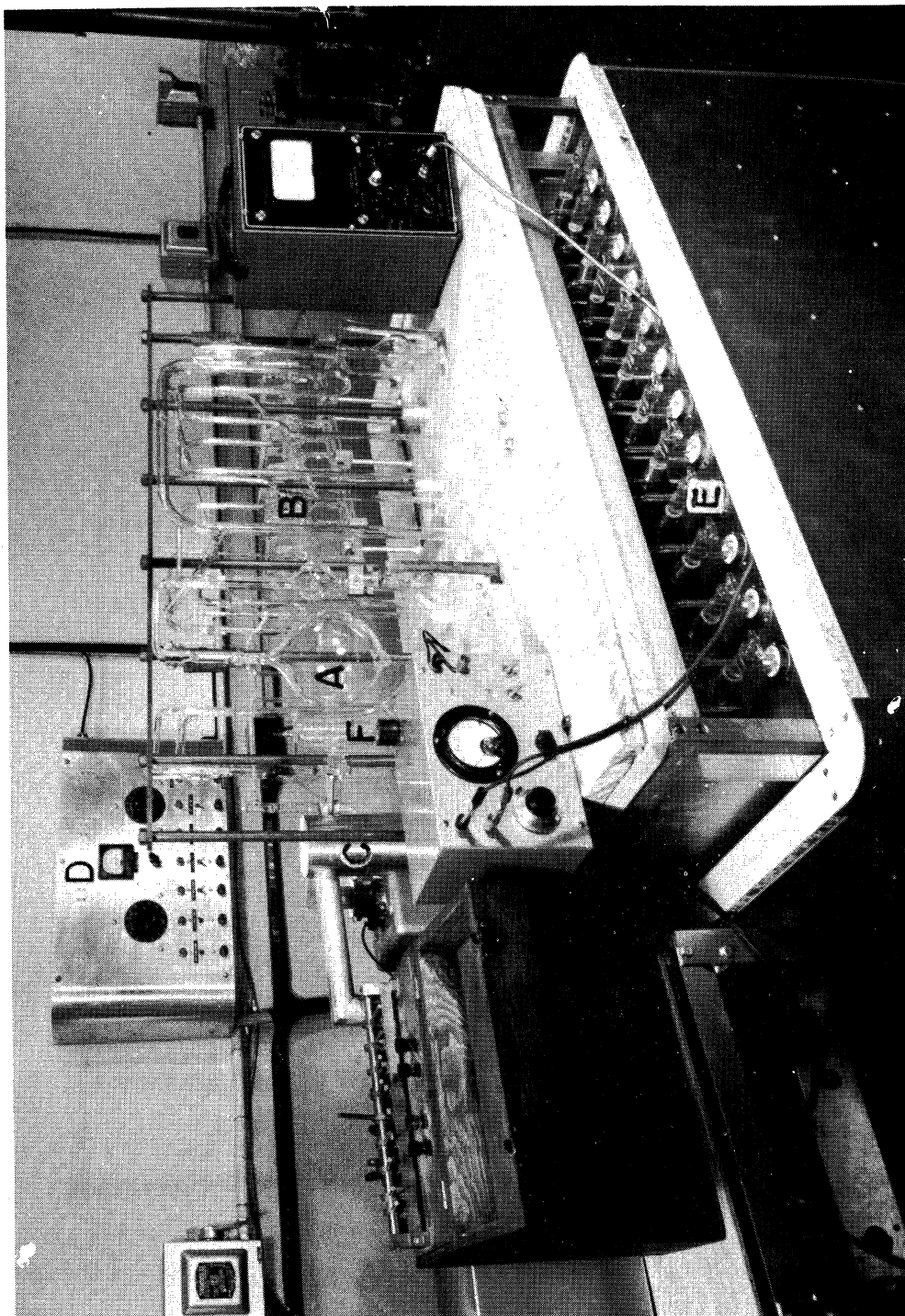


Fig. 2. New Analyzer. A. Large Toepler, B. Fractionating Column, C. Ion Gage Shield, D. Analyzer Electrical Control Panel, E. Analyzer Operating Stopcocks, F. Pirani Gage.

400-cc automatic Toepler was installed. It has worked out that after the sample source has been installed and the pump started, various tasks such as baking the charcoal traps and preparing the output gage can be carried on during pumping so that no great loss of time is encountered.

3.2 THE FRACTIONATING COLUMN

Preliminary tests showed that mercury could be successfully manipulated in capillaries 0.8 mm in diameter. The first fractionating column was constructed with tubes of this size. As a result of the glass blowing, the tubes became very slightly constricted or bent, making it extremely difficult to manipulate the mercury. A new column was constructed with 1.5-mm capillaries in critical places and 2.0-mm capillaries in places where dead volume is less important. The new column, while not a major development item, is the most intricate, difficult, and important glass component in the equipment.

3.3 THE OUTPUT GAGE

In addition to the small size and the use of soft glass, the use of an ionization gage as the output measuring device is a big departure in the design of the new analyzer. Pirani gages were used in the Durham and the first Michigan equipment. The disadvantages of Pirani gages are that they must be operated at liquid-nitrogen temperature for adequate sensitivity and because of hysteresis effects must be calibrated frequently. Also, gages suitable for the analyzer are not available commercially and must be constructed in the laboratory.

Ion gages, on the other hand, have the property of pumping the system in which they are operating. This potential drawback is avoided by operating the gage at a very low current to reduce the cleanup effect and by recording the ion current as a function of time. The latter technique permits the extrapolation of the current back to zero time, thus giving the ion current at the instant the tube was turned out. Both of these techniques have been quite successful.

Overall, the ionization gage has been applied to the analyzer successfully and its potential advantages over the Pirani gage have been realized. However, there have been, and continue to be, difficulties. These have to do with the initial outgassing and starting of the gages. Part of the trouble is due to the small-diameter tubes between the gage and the vacuum pump. In addition to this, however, there has been an inconsistency in the performance of gages of similar design (Alpert) and manufacture. Of eight gages tried, two cleaned up and pumped down with no difficulty and were used in obtaining the analyses shown below. The remainder were dif-

difficult to operate in varying degrees, a couple blackening immediately and showing practically no emission. The cause of the difficulty is being investigated.

Upon solving the various problems reviewed above (the quantitative details of which are given in previous reports), a series of analyses were carried out. A total of twelve helium analyses were performed on three samples of air and seven neon analyses on one sample of air. The results are shown in Figs. 3 and 4. The standard deviations are $\pm 1\%$ and it can be said that the analyzer is now ready to be used in measuring control and upper-air samples.

Various subsidiary experiments and investigations were performed in connection with the development of the analyzer as follows:

The distribution coefficients of carbon for helium and nitrogen were measured.

A vibrating reed electrometer for measuring small ion currents was placed in operation with the analyzer. Standard resistors for use with the electrometer were purchased and calibrated.

Pirani gages, to be used in the event that the ion gages proved unsatisfactory, were made and calibrated.

4. UPPER-AIR SAMPLES

It is planned to collect further upper-air samples in two Aerobees in the region 70-80 kilometers in order to verify the previous measurement of diffusive separation. The aerodynamic conditions during intake will be different than in previous flights to see if this has an effect on composition. A two-inch-diameter intake tube will be used on one of the three bottles of each rocket. One-inch tubes will be used on the other bottles as before. Also, the sampling will occur near peak to reduce the velocity and the ram pressure. Three major items of development were carried out in preparation for the sampling firings.

4.1 LEAK DETECTOR

A Consolidated Engineering Corporation ultrasensitive helium-leak detector Type 24-110 was purchased with Engineering Research Institute funds to be used in the preparation of bottles. This instrument is capable of detecting 1 part of helium in 2×10^6 parts of air. It has been installed on the bottle-preparation vacuum system and has successfully detected leaks of

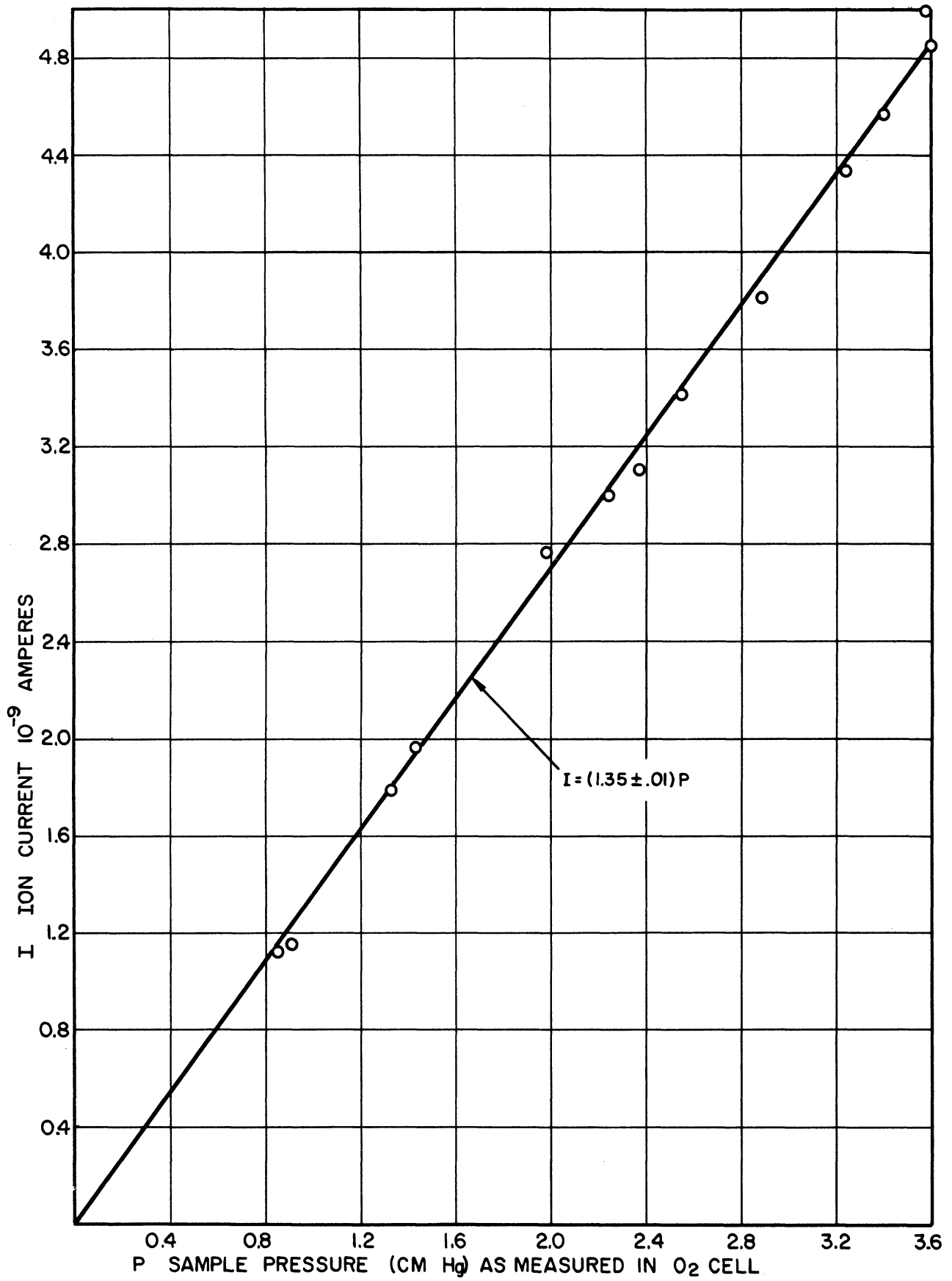


Fig. 3. Helium Analyses, Ground Air.

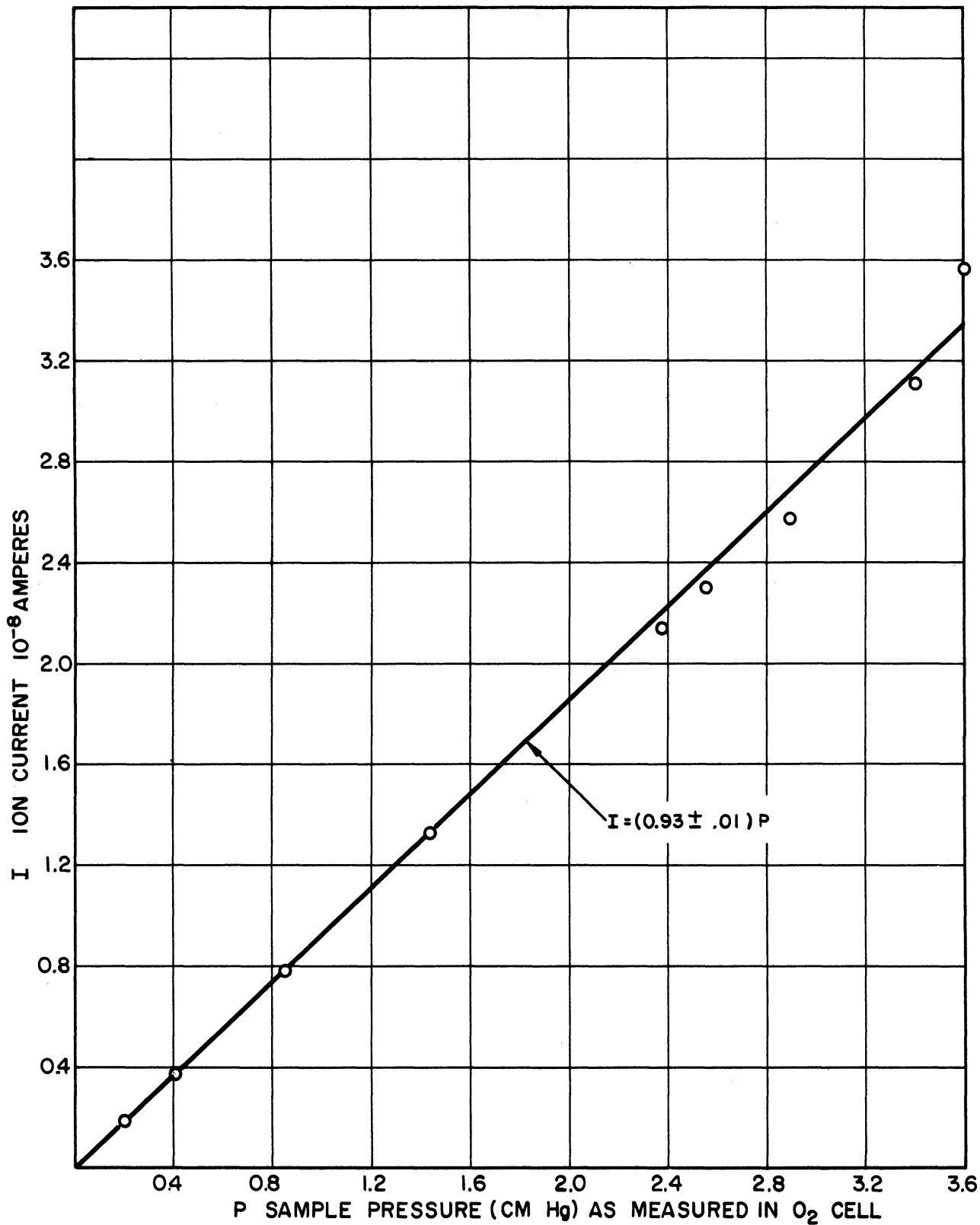


Fig. 4. Neon Analyses, Ground Air.

a size that can be tolerated in a sample bottle. A helium source delivering 2.66×10^{-6} atm cc of helium per second is used to check the leak detector.

4.2 COLD-CLOSURE AND SEALING DEVICES

Work continued on the cold-closure device described in Report 2232-8-P. The cold-closure system was developed for the purpose of opening, cleaning, and closing bottles in the laboratory during preparation and test. The system eliminates the necessity for completely rebuilding a bottle after it has been used. The cold-sealing device is used to weld shut the intake tube of the bottle after it has been opened in flight. One-inch sealers have been used successfully in flight many times and have been used successfully in the laboratory in conjunction with the cold-closure system. The two-inch sealer has been used successfully alone in the laboratory, but in each test made in conjunction with the cold-closure system an unrelated leak has prevented evaluation of the combination. It is felt that there is no inherent difficulty in the latter combination and that it will be made to work successfully.

4.3. SAMPLING ROCKET

The Aerobee instrumentation for collecting samples at peak was designed on the previous contract. A review of the design was carried out and it was decided that several simplifications and improvements were possible. Consequently, the instrumentation was completely redesigned. The salient changes are as follows:

4.31. One flight separation was eliminated. This was accomplished by inverting the bottles so that they were exposed when the instrumentation section was separated from the body of the rocket. Four springs will be used to accomplish this separation instead of an M-1 Jato.

4.32. The cutoff strip will be used in connection with the DPN-19, thus eliminating the cutoff receiver.

4.33. DOVAP will be eliminated. It was calculated that peak time could be predicted well enough to sample in the subsonic range by timer or that, alternatively, peak could be obtained from radar and sampling commanded through the cutoff strip.

4.34. The parachute section will be pressurized but the bottle section will not.

4.35. Elimination of the Jato permits simplification of the timer which previously had to accomplish two separations during the thrust of the Jato.

Figure 5 is a schematic of the new instrumentation

5. DIFFUSIVE SEPARATION

A theoretical analysis of the phenomenon of diffusive separation of a gas mixture due to combined causes of convective flow, pressure, and temperature gradients was initiated. This constitutes a continuation of a previous effort to determine whether or not diffusive separation of air can happen through the intake action of the sampling air bottle. The status of this work is the same as reported in the last report and is repeated here.

To simplify the analysis, the fluid medium is assumed to be a binary gas mixture. The object of the analysis is to determine the distribution of the concentration of one of the gas components as a function of space and time variables.

In spite of the fact that the phenomenon of diffusive separation in a flow field is involved in many physical problems, to our knowledge the theory in question has not been developed.

In comparison with the previous analysis, the compressibility effect and temperature gradient of the fluid medium are also considered.

The following analysis is restricted to one-dimensional problems.

Starting with the system of equations:

$$\frac{\partial n_1}{\partial t} + \frac{\partial}{\partial x} (n_1 w_1 + n_1 V) = 0, \text{ equation of continuity of gas "1,"} \quad (1)$$

where

- n_1 = number density,
- w_1 = diffusion velocity, and
- V = mixture flow velocity,

$$\frac{n_1 w_1}{n} = -D \left[\frac{d}{dx} \left(\frac{n_1}{n} \right) + \frac{n_1 n_2 (m_2 - m_1)}{n p} \frac{dp}{dx} + \frac{n_1 n_2}{n^2 T} \frac{\alpha dT}{dx} \right], \text{ diffusion flux equation,*} \quad (2)$$

*S. Chapman and T. G. Cowling, Mathematical Theory of Non-uniform Gases, Cambridge, 1952.

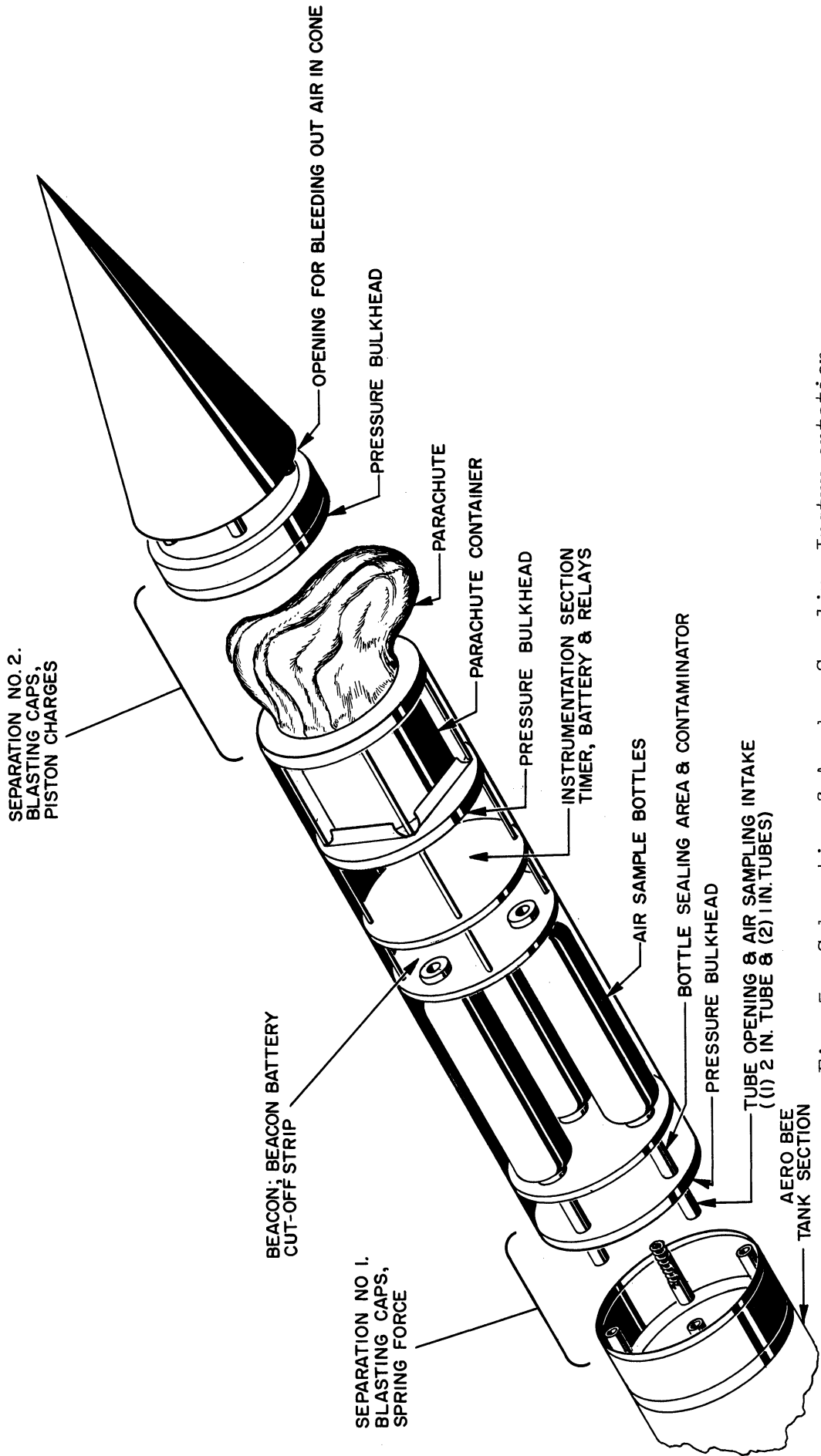


Fig. 5. Schematic of Aerobee Sampling Instrumentation.

where

- $n = n_1 + n_2,$
- $\rho = n_1 m_1 + n_2 m_2,$
- $m_1 =$ molecular masses of gas "1,"
- $m_2 =$ molecular masses of gas "2,"
- $D =$ diffusion coefficient,
- $\alpha =$ thermal diffusion factor,
- $p =$ pressure, and
- $T =$ temperature,

$$\frac{\partial \rho}{\partial t} + \frac{\partial}{\partial x} (\rho V) = 0, \text{ equation of continuity of the mixture,} \quad (3)$$

we obtain a diffusion equation which governs the concentration distribution of gas "1," namely $f = n_1/n$.

$$\frac{\partial f}{\partial t} + V \frac{\partial f}{\partial x} = \frac{\partial}{\partial x} \left[D \frac{\partial f}{\partial x} + \frac{D}{p} \frac{\partial p}{\partial x} f + \frac{\alpha D}{T} \frac{\partial T}{\partial x} f \right] + \left[D \frac{\partial f}{\partial x} + \frac{D}{p} \frac{\partial p}{\partial x} f + \frac{\alpha D}{T} \frac{\partial T}{\partial x} f \right] \frac{1}{n} \frac{\partial n}{\partial x} \quad (4)$$

assuming $m_2 \gg m_1$.

Equation 4, in conjunction with the given pressure, temperature, and convective flow velocity and appropriate boundary values of f , can be used to calculate the concentration distribution $f(x,t)$. These calculations will be made for several specific examples.

The physical significance of this analysis can be evaluated only after practical cases have been tried.

6. RECOMMENDATIONS

It is recommended that:

- 6.1. Sample bottles be prepared for flight on two Aerobeas.
- 6.2. Control experiments be run on these bottles before and after flight to detect separation due to causes other than an atmospheric phenomenon.
- 6.3. That new flight samples be obtained and analyzed at Michigan and, if possible, by Professor F. A. Paneth.

6.4. That valid residuals of previously obtained samples be reanalyzed on the new analyzer.

7. ACKNOWLEDGEMENT

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